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Effect of Stretching on Electrical Properties of Low Density Polyethylene/MgO Nanocomposites

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Abstract

Pulsed electroacoustic (PEA) method, dielectric analyzer, and high resistance meter were used to research the DC breakdown, space charge behavior, permittivity, loss tangent, and volume resistivity of neat low density polyethylene (LDPE) and LDPE/MgO nanocomposites with concentration of 1, 3, and 5 wt% with and without the elongation ratio of 1.1. Results indicate that the DC breakdown strengths of neat LDPE and nanocomposites decrease after stretching. The heterocharges near electrodes in neat LDPE change to homocharges after stretching and a large amount of positive and negative charges accumulated in samples with concentration of 3 and 5 wt%. Meanwhile, homocharges near cathode electrode in nanocomposite with concentration of 1 wt% decrease a little and a small amount of positive charges were observed in the samples after stretching. Furthermore, there are different increases in amplitudes of permittivity in all the samples after stretching, as well as the loss of tangents especially in the frequency domain from 10^{-1} to 10^2 Hz. The results of volume resistivity show that comparing with the nanocomposite with the concentration of 1 wt% whose volume resistivity decrease after stretching, larger volume resistivity is observed in neat LDPE and nanocomposites with the concentration of 3 and 5 wt%.

Keywords: stretching, LDPE/MgO nanocomposites, interface, free volume, electrical properties

1. Introduction

Polyethylene was widely used in high voltage equipment especially in DC cables because of its excellent electrical and mechanical properties [1, 2]. In comparison with the polyethylene, polymer-based nanocomposites have better electrical and mechanical properties, such

as higher DC or AC breakdown strength and volume resistivity, smaller amount of space charge accumulation, and so on [3–6]. However, polyethylene insulation materials may be affected by stretching during operation, which may lead to the change of electrical properties. Therefore, some questions arise such as: what are the properties of polyethylene and nanocomposites after stretching? And how does stretching affect the electrical properties of polyethylene and nanocomposites?

It is generally agreed that electrical properties of polymer insulation materials can improve after the addition of nanoparticles. In order to analyze the mechanism, nanocomposites with different nanoparticles and different polymer matrixes were prepared, such as LDPE/ZnO nanocomposites, LDPE/SiO₂ nanocomposites, Epoxy/TiO₂ nanocomposites, Epoxy/POSS nanocomposites, and so on [7–12]. According to the research, it is concluded that the interface between nanoparticles and polymer matrix can dominate the characteristics of polymer-based nanocomposites [13–18]. On this basis, some interface models such as multicore model and space charge model have been proposed [19–21].

Recent reports on the effects of stretching on electrical properties of polymer focus on carbon nanotube/polymer-based materials. To the best of our knowledge, there are currently no reports on the effect of stretching on the electrical properties of polymer-based nanocomposites. Previous studies have indicated that stretching can change the space charge behavior of poly(vinyl chloride) (PVC) and cross-linked polyethylene (XLPE), and even the conformations of PVC [22, 23]. In carbon nanotube/polymer composites, stretching causes deflection of the carbon nanotubes and the deflection extent can reach up to 45°, decreasing the conductivity of the carbon nanotube/polymer [24–27]. Additionally, influences of hydrostatic pressure on the dielectric properties of polyethylene/aluminum oxide nanocomposites have been researched, with results showing that permittivity and free volume will decrease after pressure [28].

Stretching may change the interface between nanoparticles and polymer matrix. This research can enhance our understanding of electrical properties of polymer-based nanocomposites. Furthermore, in order to answer the questions mentioned above, the DC breakdown, space charge behavior, permittivity, loss tangent, and volume resistivity of neat LDPE and LDPE/MgO nanocomposites with concentration of 1, 3, and 5 wt% with and without elongation ration of 1.1 were researched in this chapter. On this basis, the change of interface, space charge traps, and free volume were discussed.

2. Experiment

2.1. Material

The base polymer used was an additive-free low density polyethylene (LDPE) with a melt flow index of 2.1–2.2 g/min and a density of 0.910–0.925 mg/cm³. The nanoparticles used were MgO with a 40-nm particle size. Nanocomposites, with concentration of 1, 3, and 5 wt%, were press-modeled at 393 K and at a pressure of 10 MPa, to produce films with a thickness of about 190 μm. Samples were degassed in a vacuum drier for 7 days at 353 K. Dumbbell-shaped

samples cut by a dumbbell cutting tool were stretched in tension machine. The elongation ratio was chosen as 1.1–1.4 in the previous research [22, 23]. However, the elongation ratio of insulation material of cables may not be so high. Therefore, we choose 1.1 as the elongation ratio in this chapter. Additionally, because the polyethylene is an elastic material, the elongation ratio of samples in the tension machine should be higher than 1.1 to ensure that the elongation ratio of samples is about 1.1 after rebound.

2.2. Characteristics

2.2.1. Electrical breakdown strength testing

Samples were placed between spherical electrodes ($\phi = 6.3$ cm), which were immersed in transformer oil to avoid surface flashover on the samples. The applied DC voltage was increased linearly at a rate of 0.5 kV/s until breakdown occurred. The thickness of the tested samples was measured to calculate the DC breakdown strength. Each sample was tested 15 times and the two-parameter Weibull distribution was used to analyze the DC breakdown characteristics of the samples.

2.2.2. Space charge measurements

The space charge measurements were carried out with a pulsed electroacoustic (PEA) system, whose pulse width is 2–5 ns, pulse amplitude is 200 V, and output voltage is 0–20 kV. All samples were measured at room temperature ($25 \pm 1^\circ\text{C}$). In this chapter, a DC electrical field was applied for 1 h and the space charge formation was confirmed with the polarization of 10 s, 30 s, 1 min, 5 min, 10 min, 15 min, 20 min, 25 min, 30 min, 35 min, 40 min, 50 min, and 1 h. The reproducibility of the space charge behavior was confirmed by repeating the experiments five times for each group.

2.2.3. Dielectric properties

The dielectric properties of the materials were measured in the frequency domain from 10^{-1} to 10^6 Hz at room temperature ($25 \pm 1^\circ\text{C}$) by using a Novocontrol ALPHA-A high resolution dielectric analyzer. Prior to dielectric analysis, gold electrodes were deposited onto both surfaces of the specimens by sputtering. The diameter of sputtered electrodes is 15 mm. The results are the average measurements of five different specimens for each sample and the error in the measurement is within 2%.

2.2.4. Volume resistivity

Volume resistance was measured by a high resistance meter (model PC68). Compression-molded sheet having a diameter of 100 mm was inserted into the sample holder and charged for 1 min at 500 V. The volume resistance measurements were carried out at room temperature ($25 \pm 1^\circ\text{C}$). Volume resistivity ($\text{ohm} \cdot \text{m}$) = $21.237 (R_v/t)$, where R_v is the volume resistance (ohm) and t is the thickness (m) of the sheet. The results are the average measurements of five different specimens for each sample.

3. Results

3.1. DC breakdown

The DC breakdown properties of insulation are affected by many factors. Randomness and dispersion of DC breakdown cannot be prevented even under identical conditions. It is recognized that the two-parameter Weibull probability can fit the DC breakdown strength of solid materials effectively with the following expression [29]:

$$F(U; \alpha, \beta) = 1 - \exp\left(-\left(\frac{U}{\alpha}\right)^\beta\right) \quad (1)$$

where U represents the DC breakdown strength; α represents scale parameters, indicating the DC breakdown strength when the failure rate is 0.632; and β represents shape parameters, indicating the randomness of the data.

Figure 1 is the Weibull probability plot for the DC breakdown of neat LDPE and LDPE/MgO nanocomposites before and after stretching, and the scale parameters and shape parameters are listed in **Tables 1** and **2**.

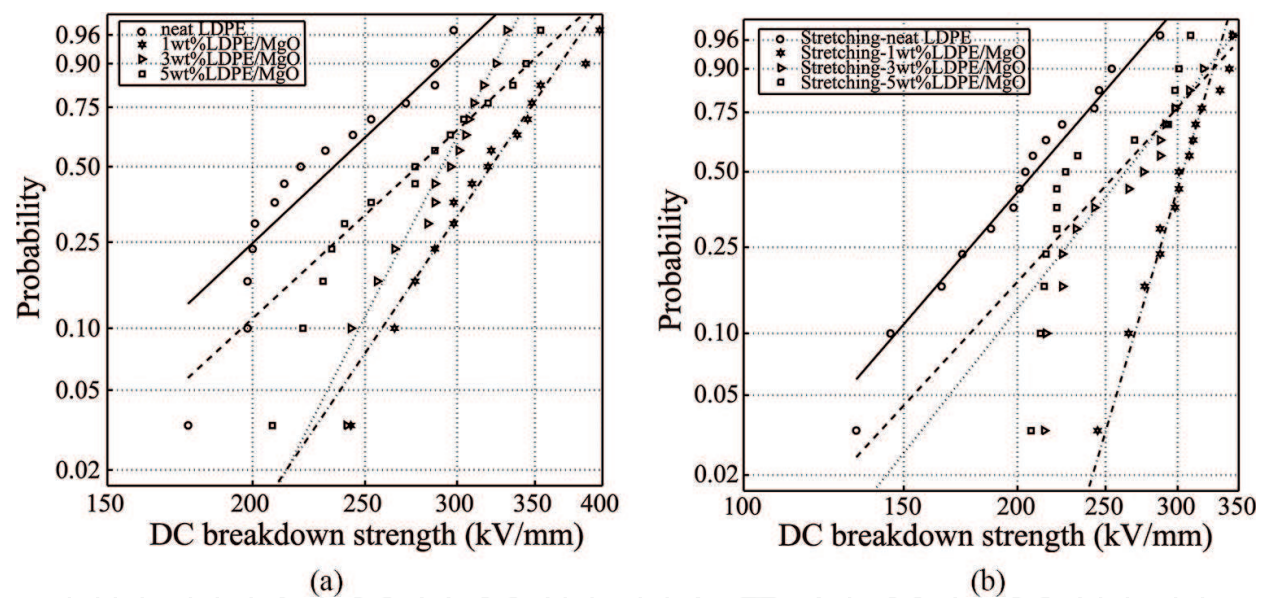


Figure 1. Weibull probability plot for DC breakdown strength of LDPE and LDPE/MgO nanocomposites before and after stretching. (a) Before stretching and (b) after stretching.

	LDPE	1 wt% LDPE/MgO	3 wt% LDPE/MgO	5 wt% LDPE/MgO
α (kV/mm)	248.76	337.71	302.07	297.92
β	6.65	8.20	13.17	6.89

Table 1. Scale parameter α and shape parameter β for LDPE and LDPE/MgO nanocomposites before stretching.

	LDPE	1 wt% LDPE/MgO	3 wt% LDPE/MgO	5 wt% LDPE/MgO
α (kV/mm)	220.59	314.39	286.05	266.32
β	5.62	12.99	7.41	6.99

Table 2. Scale parameter α and shape parameter β for LDPE and LDPE/MgO nanocomposites after stretching.

According to **Figure 1**, the scale parameters α increase after adding MgO nanoparticles, especially the nanocomposites with a concentration of 1 wt%, whose scale parameter is 337.71 kV/mm. However, the scale parameters α decrease with the increase of concentration of nanoparticles. This phenomenon is the same as the research of Murakami [30]. Furthermore, the scale parameters of all samples decrease after stretching.

On the basis of the space charge behavior, the heterocharges near electrodes in neat LDPE change to the homocharges after adding nanoparticles, which means the electrical strengths between electrodes and materials increase as well as electron injection currents decrease. Therefore, the amount of electrons or holes injected by electrodes may increase and lead to the improvement of DC breakdown strengths [31]. Additionally, the interface between nanoparticles and polymer matrix may introduce a large amount of charge traps [32]. As the carriers are trapped, the energy of nanofillers may decrease and may also promote the DC breakdown strength.

3.2. Space charge behavior

Figure 2 is the space charge behavior of neat LDPE and LDPE/MgO nanocomposites. Before stretching, heterocharges near electrodes are observed in neat LDPE with the largest amount near anode, whose value is 2.7 C/m³ after polarization time of 10 s. Meanwhile, there are positive packet-like space charges moving from anode to cathode in neat LDPE with the value of 3.5 and 3.6 C/m³ with the polarization time of 10 and 30 s, respectively. This phenomenon is the same as the research of Murakami [30]. Adding nanoparticles, homocharges are observed near electrodes in nanocomposites, whose amount increases with the increase of polarization times, especially in nanocomposites with concentration of 1 and 5 wt%. After all, all samples prepared in this chapter can suppress space charge accumulation and in which the nanocomposite with concentration of 3 wt% has the best effect.

After stretching, heterocharges near electrodes in neat LDPE change to homocharges. Space charge behavior in nanocomposite with concentration of 1 wt% changes a little and a little more positive space charges are observed. At the same time, there are positive and negative charges in nanocomposites with concentration of 3 and 5 wt% after stretching, especially in nanocomposite with concentration of 5 wt%, whose amount of positive charges is 6 C/m³ and the amount of negative charges is 7 C/m³. Interestingly, the space charge behavior of nanocomposite with concentration of 3 wt% after stretching is very similar to that of nanocomposite with concentration of 5 wt% before stretching.

It is inevitable that some residues existing in neat LDPE, which may decompose under high voltage strength, lead to the heterocharges near the electrodes [33]. On the other hand, because of

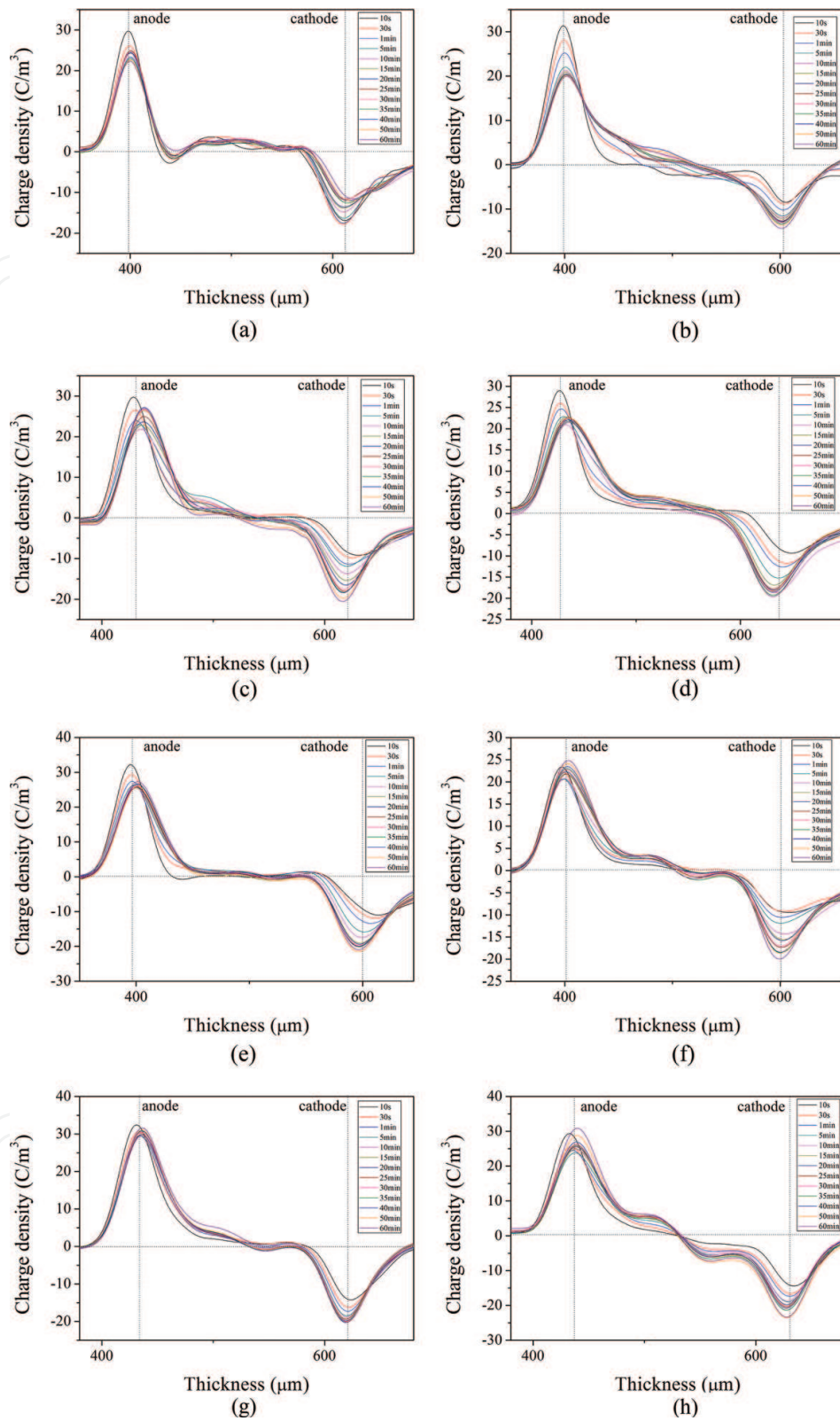


Figure 2. Space charge behavior of LDPE and LDPE/MgO nanocomposites before and after stretching during the application of a DC electric field of 40 kV/mm. (a) Neat LDPE, (b) stretching-neat LDPE, (c) 1 wt% LDPE/MgO, (d) stretching 1 wt% LDPE/MgO, (e) 3 wt% LDPE/MgO, (f) stretching 3 wt% LDPE/MgO, (g) 5 wt% LDPE/MgO, and (h) stretching 5 wt% LDPE/MgO.

the packet-like space charges observed in neat LDPE, the carriers injected by electrodes move to another electrode which may also create the heterocharges. Therefore, the heterocharges observed in this chapter may be caused by these two reasons. Due to the lack of adjacent atoms around the surface of nanoparticles, nanoparticles have high chemical activity, leading to the interface between nanoparticles and polymer matrix, which may dominate the electrical properties of nanocomposites [34]. In order to understand this interface, the multicore model has been proposed by Tanaka et al. [20]. According to this model, the interface between nanoparticles and polymer matrix can be divided into three layers: bonded layer, bond layer, and loose layer. The deep charge traps are almost distributed in bonded layer but the shallow charge traps are distributed in loose layer. Based on the research of Takada et al. [32], nanoparticle doping will increase the amount and depth of charge traps. Therefore, the carriers injected by electrodes will be trapped in charge traps leading to the homocharges in LDPE/MgO nanocomposites. On the other hand, because of the large amount of shallow traps distributed in loose layer, electrons or holes can move along the voltage field by trapping and detrapping, which means there may be overlaps between loose layers of nanoparticles as the concentration is high, such as 3 wt%, providing routes for electrons or holes and decreasing the amount of trapped carriers. This may be one of the reasons for the suppression of space charges accumulation in nanocomposite with concentration of 3 wt%. However, agglomerates may exist as the concentration is too high, such as 5 wt%, which may decrease the special surface area of nanoparticles and affect the interface between nanoparticles and polymer matrix, leading to the low capacity of suppression space charges accumulation. As shown in **Figure 2(e)** and **(g)**, there are more space charges in nanocomposite with concentration of 5 wt% in comparison with nanocomposite with concentration of 3 wt%.

3.3. Dielectric properties

Figure 3 is the dielectric properties of neat LDPE and LDPE/MgO nanocomposites before and after stretching. It is obvious that the values of permittivity of nanocomposites increase with the increase of concentration of nanoparticles, which is the same as the research of Ishimoto et al. [35]. Additionally, after stretching, the values of permittivity of all samples increase with different increasing amplitudes, in which neat LDPE has the smallest increasing amplitude but nanocomposite with concentration of 3 wt% has the largest.

Considering the large amount of free volume distributed in the interface between nanoparticles and polymer matrix, which may cause Maxwell-Wagner interface polarization [33], the values of permittivity of LDPE/MgO nanocomposites increase after adding nanoparticles. Furthermore, because the free volume mainly distributes in the loose layer of the interface, the Maxwell-Wagner interface polarization may be affected by loose layer in nanocomposites. On the other hand, the values of permittivity of nanocomposites may also increase after adding nanoparticles because of the large value of permittivity of MgO nanoparticles [35]. Additionally, according to **Figure 3(a)**, the permittivity of nanocomposite whose concentration is 5 wt% has a slope in the frequency domain from 10^{-1} to 10^0 Hz. Meanwhile, **Figure 2(g)** shows that there is a large amount of space charges in nanocomposite with concentration of 5 wt%. Therefore, it is considered that the slope may be caused by the space charge polarization in nanocomposites.

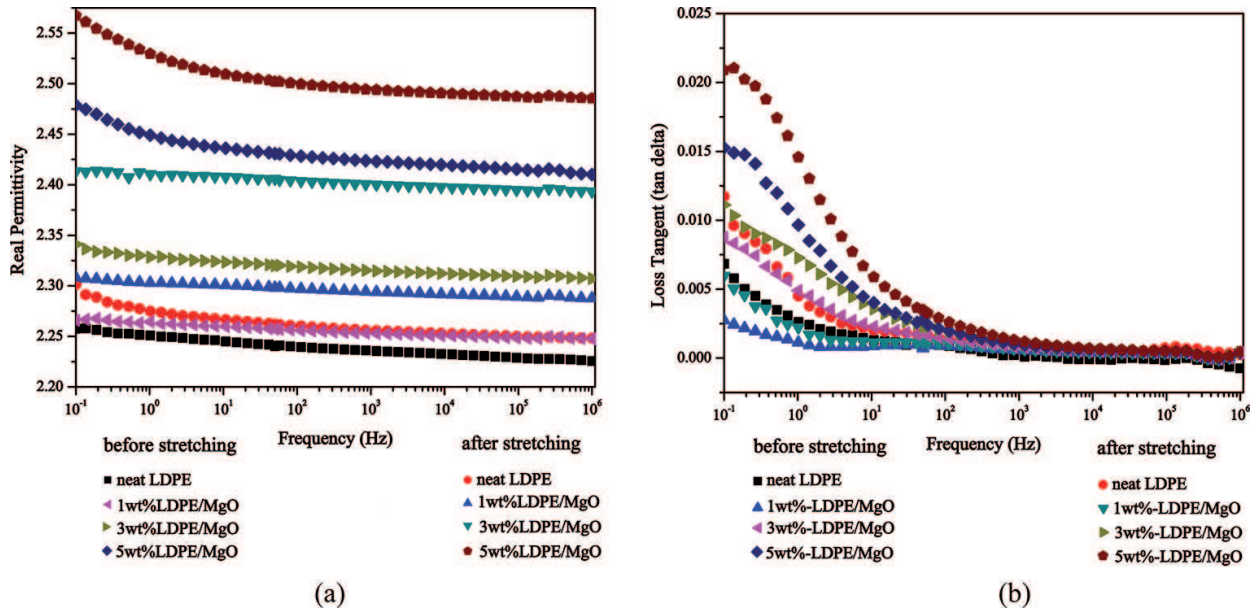


Figure 3. Dielectric properties of neat LDPE and LDPE/MgO nanocomposites before and after stretching during the application of an AC electric field of 5 V/mm. (a) Frequency dependence of real permittivity and (b) frequency dependence of loss tangent.

As shown in **Figure 3(b)**, before stretching, the gradient of slope of neat LDPE is larger than that of nanocomposite with concentration of 1 wt% but smaller than that of nanocomposites with concentration of 3 and 5 wt%. After stretching, there are little changes in the frequency domain from 10^2 to 10^6 Hz in all samples after stretching but a large increase in amplitudes in the frequency domain from 10^{-1} to 10^2 Hz, in which nanocomposite with concentration of 5 wt% has the highest value, about 0.02 at the frequency of 0.1 Hz.

Additionally, it is well known that the complicated relaxation processes of LDPE/MgO nanocomposites before and after stretching can be described by a modified Debye equation, which gives the frequency-dependent complex permittivity as follows:

$$\varepsilon^*(\omega) = \varepsilon_{\infty} + (\varepsilon_s - \varepsilon_{\infty}) / \left[1 + (i\omega \tau_0)^{1-\alpha} \right] \quad (2)$$

where α is an empirical constant with the value between 0 and 1. The case $\alpha = 0$ corresponds to the Debye model that has a single relaxation time. ε_0 is the vacuum permittivity, ε_s is the static permittivity, ε_{∞} is the dielectric constant at high frequency, ω is the angular frequency $2\pi f$, and τ_0 is the mean relaxation time. Furthermore, in **Figure 3**, the decrease of ε'' at low frequency can be attributed to the suppression of electrical conduction by the nanoparticles. Therefore, the contribution of electrical conduction to ε'' has to be considered, and the term $\sigma_0 / i\varepsilon_0 \omega$ should be added into Eq. (3), where σ_0 represents the specific conductivity and ε_0 is the permittivity of free space. Therefore, the imaginary part ε'' can be given according to the Debye function Eq. (2) by

$$\varepsilon''(\omega) = \frac{\sigma_0}{\varepsilon_0 \omega} + (\varepsilon_s - \varepsilon_{\infty}) \times \frac{(\omega \tau_0)^{1-\alpha} \cos \frac{\pi\alpha}{2}}{1 + 2(\omega \tau_0)^{1-\alpha} \sin \frac{\pi\alpha}{2} + (\omega \tau_0)^{2(1-\alpha)}} \quad (3)$$

When more than one relaxation peak exists in the polycrystalline ceramics, Eq. (3) can be rewritten as

$$\varepsilon''(\omega) = k_0 \omega^{\alpha_0} + \sum_{i=1}^n \frac{k_i (\omega \tau_{0i})^{1-\alpha_i} \cos \frac{\pi \alpha_i}{2}}{1 + 2 (\omega \tau_{0i})^{1-\alpha_i} \sin \frac{\pi \alpha_i}{2} + (\omega \tau_{0i})^{2(1-\alpha_i)}} \quad (4)$$

The first part of Eq. (4) is related to temperature-dependence electrical conductivity and the second part to the thermal polarization. The excellent agreement between the experimental data and the fitting curve based on Debye function was obtained, as shown in Figure 4.

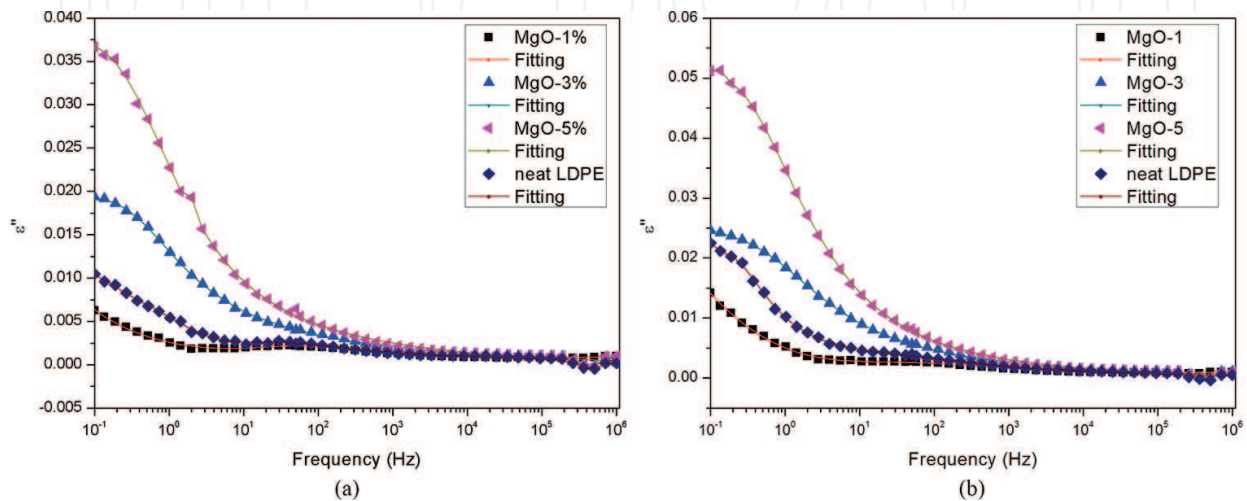


Figure 4. The representative fitting results of the dielectric loss based on Debye theory before and after stretching. (a) Before stretching and (b) after stretching.

3.4. Volume resistivity

Figure 5 is the volume resistivity of neat LDPE and LDPE/MgO nanocomposites. As shown in the image, the values of volume resistivity increase after adding nanoparticles and decrease with the increase of concentration of nanoparticles. The nanocomposite with concentration of 1 wt% has the highest value. After stretching, the values of volume resistivity of neat LDPE and nanocomposites with concentration of 3 and 5 wt% increase but decrease in nanocomposites with concentration of 1 wt%.

It is recognized that conductivity κ can be expressed by Eq. (2) [30],

$$\kappa = en\mu \quad (5)$$

where e , n , and μ represent the elementary charge, carrier density, and mobility, respectively. It is noted that the carriers are probably to be captured by traps at the interface between polymer matrix and nanoparticles in LDPE/MgO nanocomposites, decreasing the carrier mobility [35]. According to Eq. (1), the conductivity will decrease with the decrease of carrier mobility. Furthermore, when the concentration of nanofillers is high, such as 3 and 5 wt%, there are overlaps of transmission layers, increasing the carrier mobility as well as the conductivity of nanocomposites. Therefore, the volume resistivity decreases with the increase of concentration.

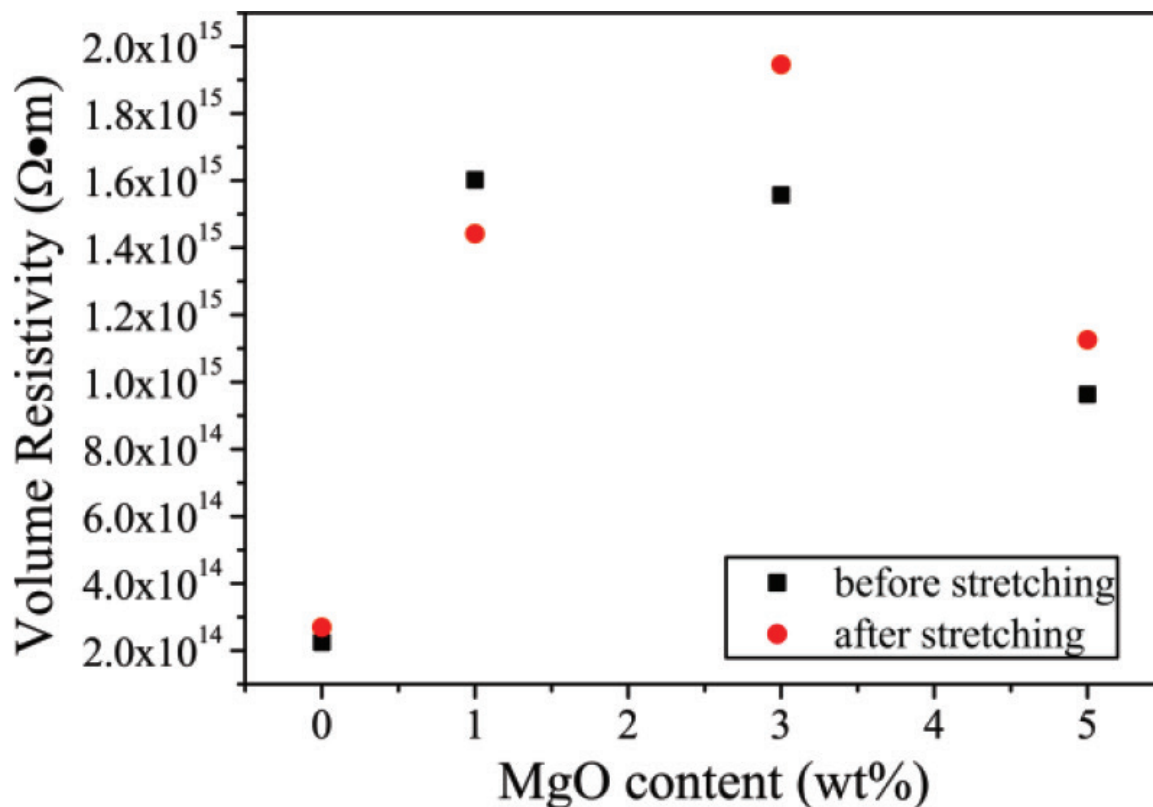


Figure 5. The dependence of MgO nanofiller content on volume resistivity during the application of a DC electric field of 2.5 kV/mm.

4. Conclusions

The effect of stretching on electrical properties of nanocomposites has been investigated. On the basis of the test results, the conclusions are as follows:

- (1) The DC breakdown strengths of neat LDPE and nanocomposites decrease after stretching, which are contrary to the volume resistivity of all samples except the nanocomposites with concentration of 1 wt%. Additionally, the values of permittivity of all samples also increase after stretching as well as the loss tangent especially in the frequency domain from 10^{-1} to 10^2 Hz.
- (2) The homocharges increase in all samples after stretching except the nanocomposite whose concentration is 1 wt% with a little more positive space charges accumulated in the sample. This may be caused by the increase of amorphous area as well as the change of interface between nanoparticles and polymer matrix after stretching.
- (3) Because of the weakness in connection between molecules in loose layer, it is considered that the area of loose layer may increase after stretching, increasing the amount and depth of charge traps as well as the free volume. The change of loose layer may be the main reason for the change of electrical properties of nanocomposites after stretching.

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